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APPENDIX

APPARENT DENSITY OF ACTIVATED CARBON

1. Weigh a 100-ml. graduate cylinder and record the weight in grams. A trip balance may be used. A vibrating shaker is attached to a ring stand above the graduate cylinder. The shaker may be obtained from the manufacturer of the activated carbon.

2. Pour a sufficient amount of the carbon to be tested in to the funnel at the top of the shaker, place the graduate cylinder under the shaker, and fill to the graduate cylinder at approximately 1 ml/sec.

3. Weigh the graduate cylinder and carbon record the weight in grams.

4. To calculate the apparent density, determine the weight of carbon in the full graduate and divide by 100

Weight carbon = Weight full graduate - weight empty graduate.

$$\text{Apparent density} = \frac{\text{Weight carbon}}{100} \text{ (gm/ml)}$$

IODINE NUMBER

The iodine number is defined as the milligrams of iodine adsorbed by one gram of carbon when the/ iodine concentration of the residual filtrate is 0.02 N.

Procedure:

1. Grind a representative sample of carbon until 90 percent or more will pass a 325-mesh sieve (by wet screen analysis).

2. Dry the sample for a minimum of 3 hr. in an electric drying oven maintained at 150 °C.

3. Weigh 1.000 g. of dried pulverized carbon (see note 2).

4. Transfer the weighed sample into a dry, glass-stoppered, 250-ml. Erlenmeyer flask.

5. To the flask add 10 ml of 5%-wt HCl acid and swirl until carbon is wetted.

6. Place flask on hotplate, bring contents to boil and allow to boil for only 30 sec.

7. After allowing flask and contents to cool to room temperature, and 100 ml of standardized 0.1 N iodine solution to the flask.

8. Immediately stopper flask and shake contents vigorously for 6 min.

9. Filter by gravity immediately after the 6-min shaking period through an E & D folded filter paper.

10. Discard the first 20 or 30 ml of filtrate and collect the remainder in a clean beaker. Do not wash the residue on the filter paper.

11. Mix the filtrate in the beaker with a stirring rod and pipette 50 ml of the filtrate into a 250-ml Erlenmeyer flask.

12. Titrate the 50-ml sample with standardized 0.1 N sodium thiosulfate solution until the yellow color has almost disappeared.

13. Add about 2 ml of starch solution and continue titration until the blue indicator color just disappears.

14. Record the volume of sodium thiosulfate solution used.

15. Calculate the iodine number as follows:

$$\frac{X}{m} = \frac{A - (2.2B \times \text{ml of thiosulfate solution used})}{\text{Weight of sample (grams)}}$$

$$C = \frac{N_2 \times \text{ml of thiosulfate solution used}}{50}$$

$$\text{Iodine Number} = \frac{X}{m} D$$

Where X/m = mg. iodine absorbed per gram of carbon

N_1 = normality of iodine solution

N_2 = normality of sodium thiosulfate solution

A = $N_1 \times 12693.0$

B = $N_2 \times 126.93$

C = residual filtrate normality

D = correction factor (obtained from Figure 19)

Notes on Method:

 1. The capacity of a carbon for any adsorbate is dependent on the concentration of the adsorbate in the medium contacting the carbon. Thus, the concentration of the residual filtrate must be specified, or known, so that appropriate factors may be applied to correct the concentration to agree with the definition.

 2. The amount of sample to be used in the determination is governed by the activity of the carbon. If the residual filtrate normality (C) is not within the range 0.008-0.035 N, given on the iodine Correction Curve, the procedure should be repeated using a different size sample.

 3. It is important to the accuracy of the test that the potassium iodide to iodine weight ratio is 1.5-1 in the standard iodine solution.

Reagents and Equipment:

 Hydrochloric acid, 5%-wt. To 445 ml of distilled water add 55 ml of reagent-grade concentrated hydrochloric acid.

 Sodium thiosulfate, 0.1 N. In a 1-l volumetric flask dissolve 24.82 g/of reagent-grade sodium thiosulfate crystals ($Na_2S_2O_3.5 H_2 O$) in distilled water. Add about 0.1 g of reagent-grade sodium carbonate and dilute to the 1-l mark. This solution should be allowed to stand for a few days before standardizing. Standardize with reagent-grade metallic copper. Dissolve about 0.2 g of copper, weighed to the nearest 0.1 mg, in 5 ml of concentrated nitric acid

and boil gently to expel brown fumes. Dilute to about 20 ml with distilled water and add ammonia water dropwise until the solution is a deep blue color. Boil again until the odor of the ammonia is faint. Neutralize with acetic acid until the precipitate which forms with the acid dissolves and add 5 or 6 drops in excess. Again bring to boiling. Cool to room temperature. Add solid potassium iodide in sufficient amount to redissolve the copper iodideprecipitate which forms. Titrate with sodium thiosulfate until the iodine fades to a light yellow color. Add starch indicator and continue the titration by adding the thiosulfate dropwise until a drop produces a colorless solution. Calculate the normality of the sodium thiosulfate as follows:

Normality of sodium thiosulfate $= \dfrac{\text{weight copper}}{\text{ml thiosulfate} \times 0.06354}$

Iodine solution. Dissolve 127 g of reagent-grade iodine and 191 g of potassium iodide in distilled water (see Note 3). Dilute to 1.1 in a volumetric flask. To standardize the iodine solution, pipette 25.0 ml into a 250-ml Erlenmeyer flask and titrate with the standardized 0.1 N sodium thiosulfate. Use the starch indicator when the iodine fedes to a light yellow color. Then finish the titration by adding the thiosulfate dropwise until a drop produces a colorless solution. Calculate the normality of the iodine solution as follows:

Normality of iodine solution $= \dfrac{\text{ml thiosulfate} \times \text{normality thiosulfate}}{25}$

Starch indicator. Mix 1 g of soluble starch with a few milliliters of cold water. Pour the mixture into 1.l of boiling water and allow boiling to continue for a few minutes. This solution should be made up fresh daily for best results.

Filter paper E & D, folded filter paper, 18.5 cm. No. 192.

ISOTHERMS

Data for plotting isotherms are obtained by treating fixed volumes of the liquid to be tested with a series of known weights of carbon. The carbon-liquid mixture is agitated for a fixed time at constant temperature. After the carbon has been removed by filtration, the residual organic content of the solution is determined. From these measurements, all of the values necessary to plot an isotherm may be calculated.

The isotherm tests may be performed at room temperature unless the anticipated plant operation will be at a significantly different one. To determine the need contact time for the isotherms, a preliminary experiment should be run in which fixed volumes of the wastewater are contacted with a fixed weight of carbon for 1,2, 3 and 4 hr. periods. A contact time sufficiently long to insure a reasonable equilibrium should be chosen from these data for the isotherms.

The p^H of the wastewater being tested will affect the carbon efficiency. Care should be taken to insure that the p^H of the test sample is representative of anticipated plan-scale conditions.

In order to minimize the variable of carbon particle size in the isotherm test, the granular carbon should be pulverized so that 95 percent passes a 325 mesh screen. If suitable mechanical equipment, such as a ball mill, is not available to pulverize the carbon, a mortar and pestle can be used, although this is a tedious approach. the manufacturer of the carbon may also be able to supply pulverized material.

For the initial isotherm test, carbon dosages of 50, 100, 150, 200, and 300 mg/l. of wastewater may be used. If the highest dosage is not adequate to effect the desired degree of treatment, higher dosages should be tried until that degree is achieved. If one of the intermediate dosage is sufficient to effect complete or satisfactory removal one or two lower dosages should be tried. In order to obtain a satisfactory isotherm, as wide a range of organic removal should be obtained as is practical.

Isotherm Procedure:

1. Pulverize a represnentative sample of the granular carbon (a 10-20 g sample is usually adequate) so that 95 percent will pass through a 325-mesh screen. Oven-dry the pulverized sample for 3 hr. at 150°C .

2. Obtain a representative sample of the wastewater to be tested. Suspended matter should be removed by filtration.

3. Transfer four different weights of the oven dried pulverized carbon to the test containers. Stoppered flasks or pressure bottles are satisfactory containers.

4. To one container, add 100 ml of wastewater from a delivery burette or graduate cylinder, and clamp the container on a mechanical shaker. The samples must be constantly agitated during the isotherm test and a mechanical shaker is desirable. A Burrell Wrist Action Shaker, Catalog No. 75-775, is satisfactory. Agitate the mixture for the chosen contact time. The bottles may be filled and placed on the shaker at 10 or 15 min. intervals to give the analyst sufficient time to filter each sample immediately after the the contact time has elapsed. The same volume of wastewater should be added to a container without carbon and subjected to the same procedure /order to obtain a blank reading:

5. After the chosen contact time has elapsed, filter the contents of the flask through either a laboratory pressure filter fitted with an asbestos disk or through a Buchner funnel containing a filter paper inserted in a filter flask connected to a vacuum. The blank should be filterred in the same manner as the other samples. It is desirable to discard the first and last portions of the filtrate and save only the middle portion for analysis.

6. Determine the organic content of the filtrate.

7. Tabulate the data as shown in Table 17. The residual solution COD concentration, C, is obtained directly from the filtrate analysis.. The amount adsorbed on the carbon, X, is obtained by subtracting the value of C from that Co, the influent concentration. Dividing X by M, the weight of carbon used in the test, gives the amount adsorbed per unit weight of carbon.

8. On log paper plot C on the horizontal axis against X/m on the vertical axis and draw the best straight line through the point as illustrated in Figure 19.

TABLE   17   TABULATION OF ISOTHERM DATA

M weight of carbon (mg/100 ml) solution	C Residual COD (mg/l)	X COD Adsorbed (mg)	X/m COD Adsorbed per unit weight
0	40	-	-
5	27	1.3	0.26
10	19	2.1	0.21
15	14	2.6	0.17
20	10	3.0	0.15

From the isotherm, it is immediately apparent whether or not the desired degree of purification can be attained with the particular activated carbon tested. If a vertical line is erected from the point on the horizontal scale corresponding to the influent concentration (Co) and the isotherm is extrapolated to intersect that line, the X/m value at the point of intersection can be read from the vertical scale.

This value, $(\frac{X}{m})$ Co, represent the amount of COD adsorbed per unit weight of carbon when that carbon is in equilibrium with the influent concentration. Since this should eventually be attained during solution treatment, it represents the ultimate capacity of the carbon. However, experience has shown that it is difficult to accurately predict the carbon dosage from isotherm tests for plant-scale carbon column. Pilot carbon column tests conducted over several weeks are the only accurate means of determining the required carbon dosage.
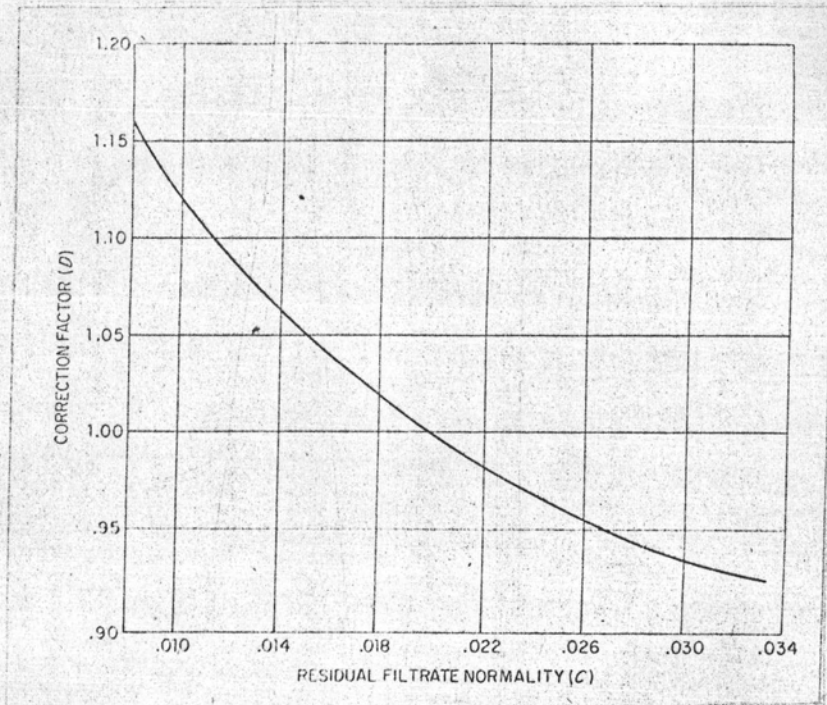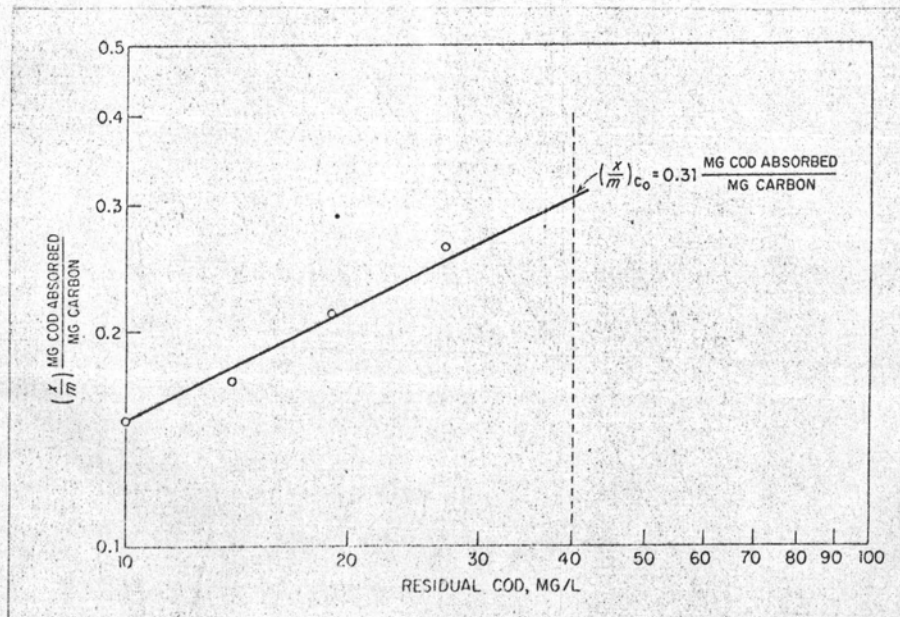
FIGURE 19 IODINE CORRECTION CURVE

FIGURE 20 ILLUSTRATIVE ISOTHERM

VITA

The author, Somchai Ekthamsut, was born on August 28, 1949 in Nakornpathom, Thailand. He received a Bachelor's Degree of Engineering (Second Class Honours) in Civil Engineering from Chulalongkorn University in 1970. At the time writing the thesis, he is serving as the government official of Location and Design Division, Department of Highways.